PICATINNY ARSENAL

TECHNICAL DIVISION

TECHNICAL REPORT

SUBJECT: ADHESIVE AND SEALER PROBLEMS (POLYESTER SEALERS FOR THREADED METAL PARTS)

PROJECT NO. TB4-621 REPORT NO. 1

PREPARED BY: S. S. Stivala DATE: July 1951

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ADHESIVE AND SEALER PROBLEMS

(POLYESTER SEALERS FOR THREADED METAL PARTS)

Project No. TB4-621 Report No. 1

Picatinny Arsenal Technical Report No. 1856 July 1951

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PICATINNY ARSENAL

Authorization: Ordnance Research and Development Division, ORDTB

Project No. TB4-621 Report No. 1

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Project Title: Adhesive and Sealer Problems (Polyester Sealers for Threaded Metal Parts)

OBJECT

To develop a series of sealers of varying strengths for use in fastening together threaded metal parts.

SUMMARY

A series of sealers has been developed for fastening threaded metal parts together. It appears that these sealers will be widely usable in place of set-screws, lock washers, staking, and other techniques and devices for preventing the unscrewing of assembled parts. The sealers are unaffected, or only moderately affected, by moisture, temperature changes from -65°F to +160°F, and vibration, but the sealer can be chosen so that subsequent unscrewing and disassembly will be possible at practically any specific torque which is desired. The sealers set at room temperature in about 3 to 4 hours. All materials used are commercially available.

CONCLUSION

Seven polyester and polyester/plasticizer compositions provide thread sealers by which threaded parts can be sealed together with a variety of degrees of strengths. With these sealers, parts can be held together with great firmness or with practically any desired lesser degree of firmness. The sealers harden at room temperature within a few hours and show good retention of properties under extreme temperature, humidity, and vibration conditions. It would appear that these sealers will have a wide utility in Ordnance applications.

Since these sealers will not harden in contact with copper, brass, or lead, or in contact with rubber compositions which contain sulfur, the sealers must be used between surfaces which do not consist of these materials.

RECOMMENDATIONS

1. It is recommended that these sealers be used for fastening together threaded metal parts in general accordance with the information provided in this report. Appendix 1 contains condensed information on the selection, application, and use of sealers, this Appendix being specifically intended as a brief manual on these materials for the use of design engineers.

2. It is recommended that a specification for these materials be prepared.

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INTRODUCTION:

1. This investigation was undertaken in an effort to develop an improved method for fastening threaded parts together securely. Setscrews, lock washers, and staking have been widely employed for this purpose in the past. Setscrews and lock washers are not applicable in all instances because of space limitations and inaccessibility. Staking is permanent but must be skillfully done and will not permit subsequent disassembly if the need arises.

2. Accordingly, it could be foreseen that a liquid sealer or a series of liquid sealers would have many useful applications. Preferably, the sealers in the series should vary in strength so that the torque required for subsequent disassembly could be chosen at will.

RESULTS:

3. The results on the study of polyester compositions as thread sealing materials are recorded in Tables 1 to 9, inclusive. Figures 1 to 9 present the same data in the form of graphs. Results obtained can be summarized as follows:

a. Effect of type of resin--Polyester A (rigid type) produced a higher torque than did Polyester B (flexible type). Torques of approximately 450 ft-lbs were produced with Polyester A on 1-inch-diameter bolts, and torques of about 240 ft-lbs were produced by Polyester B. These data are shown in Table 1.

b. Effect of catalyst--Varying concentrations of catalyst from ½% to 4% did not appreciably alter subsequent torque. These data are shown in Table 2, and in Figure 1.

c. Polyesters modified with liquid polybutadiene--Polyesters modified with liquid polybutadiene produced appreciably lower torques than unmodified polyesters. This was true for both the rigid and flexible type polyesters. The decrease in torque was most marked at low concentrations of polybutadiene, concentrations above 1% producing proportionately less reduction in torque. This is shown in Tables 3 and 4, and in Figures 2 and 3.

d. Polyesters plasticized with dimethyl phthalate--Dimethyl phthalate as a plasticizer was relatively ineffective with Polyester A but was very effective in reducing the torque produced by Polyester B. One to five percent dimethyl phthalate incorporated with Polyester A did not produce much decrease in subsequent torque, although 10% or more dimethyl phthalate did produce an appreciable decrease. The above data are shown in Table 5A and in Figures 4 and 5.

e. Polybutadiene as compared with dimethyl phthalate--Polybutadiene is more effective in reducing torque than is dimethyl phthalate. For any given concentration, polybutadiene produced a greater drop in torque than did the dimethyl phthalate. This is shown in Figure 7.

f. Polyesters plasticized with ethyl lactate - The behavior of ethyl lactate as a plasticizer is similar to that of dimethyl phthalate. Ethyl lactate employed with Polyester B effectively decreased torque; whereas, ethyl lactate with Polyester A was relatively ineffective at percentages less than about 10%. This is shown in Table 9.

g. Polyesters filled with carbon black - Concentrations of 1/2% and 1% carbon black with either Polyester A or B failed to lower torque. This is shown in Table 8.

h. The effect of bolt diameter on torque resistance - For any given resin, the torque on a 1-inch-diameter bolt is greater than on a 1/2-inch-diameter bolt. In general, the increase in torque is approximately proportional to the increase in the exposed sealing area and the increased moment arm. This is shown in Figure 8.

i. Effect of vibrations - Vibrating bolts initially at -65°F and at +160°F did not appreciably alter the torque of any of the four compositions, i.e., pure Polyester A, pure Polyester B, Polyester B plus 10% dimethyl phthalate, and Polyester B plus 10% polybutadiene. The results of the vibration tests are shown in Table 6 and in Figure 9.

j. Effect of low and high temperature on torque - Assemblies tested at -65°F produced higher torque than did assemblies tested at room temperature; whereas, assemblies tested at +160°F produced lower torques. The results of high and low temperature tests are shown in Tables 5 B and C and in Figure 6.

k. Effect of temperature cycling - When bolts sealed with the compositions listed under "Effect of vibration" were subjected to temperature cycling between -65°F and +160°F, the torques obtained were essentially the same as for the uncycled samples. This is shown in Table 7 and Figure 9.

DISCUSSION OF RESULTS:

4. Among the various materials available for examination as thread sealers, polyesters possess several advantages. In particular, polyesters are available which are thermosetting at room temperature, and polyesters are known to be relatively stable in properties under the adverse conditions of storage to which completed Ordnance items are often exposed. In addition, polyesters are available with a wide range of properties.

5. Briefly stated, polyesters are condensation products resulting from the reaction of polybasic acids and polyhydroxy alcohols with consequent elimination of water. The reaction of a saturated dibasic acid and a saturated dihydric alcohol will yield a linear polymer. Since there are no unsaturated groups in the chain, subsequent cross-linking is not possible and, thus, the polymer is permanently thermoplastic. However, the most widely used polyesters contain a significant portion of malic acid or some other constituent which has an ethylenic bond after condensation.

6. To this linear, partially unsaturated polyester, is added a vinyl monomer of some sort, generally styrene. The principal purpose of the styrene is to convert the original polyester into a cross-linked, three-dimensional, thermoset polymer. This conversion takes place only after addition of peroxide catalyst to the polyester/styrene mixture. Incidentally, the monomeric styrene serves two other useful purposes: (1) the styrene reduces the cost of the final polyester, and (2) the styrene lowers the viscosity of the mixture previous to final polymerization. During the final polymerization-cross-linking between the original unsaturated polyester and the styrene - the reaction involves only molecular addition, and there is no elimination of water. Accordingly, polyesters differ significantly from other thermosetting resins, such as the phenolics, in that the latter resins cure by condensation, thereby liberating water which, thus, demands high temperatures and pressures.

7. All polyesters follow a general pattern during the final curing or polymerization process. The liquid polyester is first converted to a gel. Gelation is then almost immediately followed by further polymerization to the final infusible solid. It is during the last stages of polymerization that all the ultimate physical properties of the polyester are developed.

8. Most polyesters can be cured at room temperatures in a relatively short time. The resins used in this investigation generally cured within three to four hours when catalyzed with 1% methyl ethyl ketone peroxide by weight.

9. Thus far, the discussion has consisted of a brief description of polyesters in general. The main objective of this investigation, however, has been the selection of a material capable of mechanically sealing threaded parts together.

10. Two possible mechanisms are immediately apparent by which polyesters might provide the binding action which is observed. In the first place, the polyesters might bind the bolts in place by the adhesive action of the polymers. In the second place, the polyesters might bind the bolts in place simply by filling and obstructing the threads. On the basis of observations to date, it is believed that the first of these possible mechanisms plays only a minor part in the observed performance and that the binding action is probably due principally to the second mechanism.

11. The first mechanism seems improbable because polyesters have relatively poor adhesion to steel. In addition, lower temperatures would not be expected to increase torque and would, if anything, result in separation of the sealer from the metal and, hence, would result in lesser torque.

12. If, on the other hand, the polyester simply fills and obstructs the threads, the resisting torque which results would be a function of a great many variables, most of which so far have not been measured, but which would include the shear strength of the polyester. As far as the available data go,

the data are consistent with this explanation since the higher strength polyesters have produced the greater binding torques. As a result, this is a tenable explanation for the mechanism involved even though the data collected are insufficient to prove that this is unquestionably the mechanism which is involved or that this is the sole mechanism.

13. The observed variation in torque with variation on bolt diameter provides no indication of the mechanism by which torque resistance is produced because the variation observed is in accord with expectations in either case. Whether produced by shear strength of the polyester or by adhesion, torque would be expected to be proportional to the area of sealer and to the moment arm of that area about the center of rotation. Taking these into consideration, the torque resistance produced in 1-inch diameter bolts would be expected to be about eight times that produced on 1/2-inch diameter bolts. Observed torque values are very close on this ratio.

14. From the foregoing investigations of polyester sealers applied to bolts, it is apparent that the torque produced is determined by several significant variables. These variables include both the characteristics of the material which is used as sealer and the characteristics of the bolt and threads which are being sealed.

15. The relevant properties of the material used as sealer depend on the properties of the cured polymer itself and upon the modification in characteristics which is introduced by adding plasticizer. The relevant properties of the bolt and thread system consist of the following: (a) diameter of the bolt, (b) class of threads, (c) type of threads, (d) knicks and irregularities that are found in threads due to machining, and (e) the contour and other dimensional characteristics of any threaded piece.

16. Effect of catalyst. - To initiate the final polymerization or cure, catalysts are used. These are usually of the free radical producing type, such as peroxides. The catalyst, to some degree, contributes to the ultimate property of the final resin by virtue of its ability to control chain length. In this study, effect of catalyst concentrations was investigated using concentrations of 1/4% to 4%, by weight. For all practical purposes it can be concluded that catalyst concentrations will not appreciably alter the torque.

17. Effect of properties of the sealer - It was mentioned above that the torque produced seems to be mainly due to mechanical obstruction of the threads. Consequently, it follows that a resin having high mechanical strength under compressive and shear stresses will produce high torque values. This was found to be the case, since resins ranging from the most rigid to the most flexible yielded torques of decreasing magnitude with decreasing rigidity; with polymers of this sort compressive and shear strengths generally decrease with decreasing rigidity and hardness. It will be seen that rigidity and hardness in themselves probably have no effect on the torque which is produced, but these two properties simply parallel roughly the strength properties which actually produce the torque resistance.

Accordingly, it is not surprising to observe that decrease of torque is not a linear function of decrease in rigidity. Actually there seems to be a sharp drop in torque at the first decrease in rigidity. The most marked drop in torque usually coincides with the first small percentage addition of polybutadiene or dimethyl phthalate, as shown in Figures 2 to 5 inclusive.

18. The use of plasticizers - An avowed objective of this work was provision of a series of sealers such that bolts could be fastened in place with various degrees of firmness and torque resistance. This has resulted in the specifying of seven sealer compositions to provide torque resistance from very high to very low. It would probably have been possible to locate seven different polyesters, of appropriately varying strengths, to cover this range. However, essentially the same could be accomplished by using fewer polyesters but modifying these with added plasticizer. The advantages in procurement, storage, and utilization seemed in favor of few polyesters with modification by varying percents of one plasticizer.

19. It should be pointed out that plasticizers are generally added to polymers to increase toughness and/or flexibility, and the concomitant decrease in strength is accepted as an unavoidable simultaneous effect. On the other hand, in the case of these sealers, plasticizers were added deliberately for the purpose of weakening the polymer.

20. When polybutadiene was used as the plasticizer, it worked well with either the rigid or the flexible resin. It is conceivable that the addition of polybutadiene served not only as a weak filler in the mechanical sense but also to some small degree as an unsaturated compound capable of entering into the polymerization reaction. Apparently, in the curing reaction, the polybutadiene modified the properties of the pure resin into a material that was weakened by introducing a longer chain cross-linking agent.

21. Dimethyl phthalate and ethyl lactate similarly weakened the polymer, but only in the case of the flexible type resin. Both of these materials are plasticizers capable only of plasticizing certain polymers. Polyester B is believed to be derived from adipic acid while, on the other hand, the rigid type polyester is believed to be derived from phthalic acid, and, in general, polymers derived from phthalic acid seem to be less capable of plasticization than those derived from aliphatic acids. Accordingly, it is postulated that in the case of polybutadiene, this material produces weakening by copolymerization with the polyester to a limited degree; whereas, in the case of ethyl lactate or dimethyl phthalate, these materials act solely as plasticizers.

22. Diameter of bolts - It is to be expected that increasing the diameter of the bolt will increase the torque required to break the seal and initiate unscrewing, both because increasing the diameter increases the area of sealer and also because increasing the diameter increases the moment arm by which the sealed area resists turning.

23. Class of threads - Most common threads are produced in classes varying from the most loose fit or Class 1 to the most tight fit or Class 4 or 5. Class 2 threads were used in this investigation and other classes were not tried. Accordingly, these results and conclusions are valid for Class 2 fits and would have to be modified in connection with other fits in accordance with subsequent investigations. Class 1 is usually encountered in large production work where speed is of prime importance. Classes 2 and up are usually used in those types of work where fit is of greater importance.

24. Type of threads - The type of thread refers to the number of threads per inch. As stated in most thread circulars, two types are accepted; namely, National Coarse and National Fine, the latter having more threads per inch. National Coarse threads were used in these experiments. Accordingly, the results and conclusions are valid in detail for this type of thread and may have to be modified on the basis of experiments with National Fine threads.

25. Up to this point the discussion has consisted of the variables affecting initial torque. Other factors can also affect retention of torque resistance and hence would affect durability as distinct from initial strength. These factors consist of variables such as weather, temperature, vibration, etc.

26. Effect of low and high temperatures - Most polymeric materials, when exposed to low temperatures, such as -65°F, become increasingly strong as well as increasingly hard and brittle. Accordingly, as would be expected, higher torque resistance was observed at low temperatures. Also, it will be observed that at low temperatures all the polymeric compositions evaluated tended to approach the same torque resistance.

27. At the other end of the scale, most polymeric materials, when exposed to higher temperatures, such as 160°F, tend to become weaker and softer. This is definitely the case for thermoplastic materials, and to a lesser degree is also true of thermosetting materials, the degree of weakening depending upon the chemical constitution of the polymer. Since the polyesters considered in this investigation are thermosetting, reduction of stress will not be extreme. As a result, the torque obtained at 160°F for any given bolt and resin was appreciably lower than at room temperature but was not so low as would be expected if a thermoplastic material had been used.

28. It was observed that as the temperature increased, the reproducibility of torque values decreased as is shown in Tables 10 and 11. Since torque is produced partly by mechanical irregularities even in the absence of any sealer, this observed increase in erraticness may be partly due to the increasing dominance of mechanical irregularities in torque production as the sealer becomes less important.

29. Effect of vibration: Severe vibrations can in many instances cause polyester pieces of the rigid type to crack. Since vibration of one form or another is encountered in many thread applications, this condition was investigated. As discussed above under the "Effect of High and Low Temperatures", it is clearly seen that extreme temperatures can alter the physical strength of most polymeric materials. In order to combine the most disadvantageous temperature condition with the disadvantageous effect of vibrations, the bolts were vibrated at extreme initial temperatures with gradual return to equilibrium or room temperatures. Since at -65°F the resin tends to become hard and brittle, it would seem that severe vibration at low temperature would have more of a detrimental effect on the resin than if severely vibrated at room temperatures. Accordingly, two sets of bolts were vibrated, one initially at -65°F and the other initially at 160°F. Further, four resin compositions were studied to determine which would be more affected under such adverse conditions.

30. Within experimental limits and for all practical purposes, it can be stated that adverse conditions of continuous vibration do not generally alter the torque for any given bolt and resin appreciably. However, this is not the case for the resin composition consisting of flexible polyester plus 10% polybutadiene. For this composition, it was observed that vibration at either extreme temperature reduced the torque by approximately 50% of the untreated bolt.

31. Effect of temperature cycling-- Since many threaded parts are exposed to varying conditions of weathering, a series of bolts sealed with four different resin compositions was subjected to temperature cycling, consisting essentially of exposing the bolts to -65°F for 8 hours followed immediately by 160°F and 90% R.H. for 16 hours. This was repeated for a period of six days. Under such adverse conditions the bolts were found to be totally unaffected.

32. Contaminants-- One precaution must be kept in mind in connection with the curing of polyesters. These materials will not cure along surfaces which are in contact with copper, brass, or lead or in contact with rubber compositions which contain sulfur. Accordingly, the sealers would not cure if used with brass bolts. If, for example, rubber or lead washers were used in conjunction with steel bolts, the polyester would remain sticky and would not cure where in contact with the washer, but would be expected to cure normally in the threads slightly removed from the washer.

33. Summary-- It has been found that polyesters provide remarkable and controllable resistance to torque in threaded parts. It seems that this application of polyesters can eliminate the use of self-locking threads, washers and other mechanical devices in many types of assemblies. The stronger polyesters produce very high torque, making the removal of a 1"-diameter bolt very difficult. In some intended applications this is desirable, while in others a somewhat different result may be preferable. Considering a specific application, it may be desired that a threaded part be capable of withstanding a limited, specified torque, but be capable of unscrewing under a somewhat higher torque. Such an application might occur in any machine assembly, in which the sealer would prevent loosening from vibration but would permit unscrewing under the action of a wrench. The degree of torque resistance required can be obtained by varying the composition of the polyester sealer.

EXPERIMENTAL PROCEDURE:

34. Resin preparation--Essentially, the procedure of preparing the resins consisted in mixing the pure resin and the catalyst by hand-stirring. All resins were catalyzed with 1% methyl ethyl ketone peroxide, which is the percentage by weight of the resin or in some cases of the total weight of the resin polybutadiene, etc. All percentages recorded are on a weight basis.

35. Resin application--Application of resin to bolts was accomplished by spreading the resin onto the bolts by means of a brush. In some cases, dipping the threaded ends of the bolts into a beaker of resin was also used. The nuts were screwed onto the bolts until the end of the screw and the lower face of the nut were flush. Bolts were than allowed to stand at room temperature for at least one day before any testing was conducted.

36. High and low temperature testing--Bolts that were subjected to high and to low temperature were stored at the respective temperatures for a period of 24 hours. Torque was determined by means of torque wrenches. Due to the lack of proper facilities, one bolt at a time was removed from the conditioning boxes and immediately tested at room temperature on a vise. It is believed that no appreciable change in temperature would take place in testing the bolt by such means since only a short interval of time is required for the actual testing of torque.

37. Cycling--Bolts were subjected to -65°F for a period of eight hours. This was immediately followed by subjecting the bolts to an atmosphere of 160°F and 90% R.H. for 16 hours and than followed by another 8 hours at -65°F. This hot-cold treatment was performed for six consecutive days. Torque was then determined at room temperature.

38. Vibration--One set of bolts was stored at -65°F for 24 hours and a second set was stored at 160°F, 90% R.H., also for a period of 24 hours. Both sets of bolts were placed on a mechanical vibrator, having 550 rpm and $\frac{1}{4}$ "eccentric", and were vibrated at room temperature for four hours. The bolts started at -65°F or at +160°F, but came to room temperature during the vibration. The bolts were tested for torque at room temperature.

39. Condition of tests--Unless otherwise specified, all bolts were tested at room temperature. Two torque wrenches were in use, one having a capacity of 600 in-lbs for the $\frac{1}{2}$-inch-diameter bolt and the other having a capacity of 600 ft-lbs for the 1-inch-diameter bolts. Further, the 1-inch-diameter bolts were of the National Coarse, Class 2. The $\frac{1}{2}$-inch-diameter bolts were also of the National Coarse, Class 2 classification.

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CODE SHEET

For use with Picatinny Arsenal Technical Report No. 1856

Material	Commercial Designation	Source
Polyester A	"Laminac" 4116	American Cyanamid Company
" B	" 4134	" " "
Methyl Ethyl Ketone Peroxide	"Lupersol" DDM	Novadel-Agene Corporation

Appendix 1

Choice and Preparation of Polyester Thread Sealers for Ordnance Applications

Choice of Composition

Choice of composition will depend upon the torque resistance which is desired. Tables 10 and 11 show the torque values which can be expected with 1"-diameter bolts and 1/2"-diameter bolts with 7 different sealer compositions. Additional intermediate values could be obtained with corresponding intermediate compositions. However, the torque values listed are believed to cover relatively well the range that will generally be required, and it is recommended that some one of these 7 compositions be specified whenever possible.

In these tables, the variations shown will include 99% of the values that will be encountered. These predictions are based on a statistical analysis, this analysis being dependent on the distribution as expressed by the standard deviation. Since the number of samples available were relatively small, and since the method of statistical analysis employed did not include a factor for consistent trends, the predicted variations are overly large if anything and would not be expected in any case to be found subsequently to be smaller than should have been predicted.

Preparation of Sealer

The sealer composition will consist of polyester resin to which catalyst has been added to which plasticizer may have been added. The polyester should be weighed with an accuracy of ± 1% of the weight involved, and the catalyst and plasticizer should be weighed with an accuracy of ± 5% of the weight involved. The plasticizer and catalyst can be added to the polyester and all three then mixed. Mixing should be sufficient to secure uniform distribution of both plasticizer and catalyst throughout the whole.

Application of Sealer

The catalyzed composition is applied to threaded parts by brush or by dipping the end of the bolt in a container of the thread sealing composition. In either case the composition is applied to the bolt only.

Curing

Polyester A will harden within approximately 4 - 6 hours at 77°F.

Polyester B will harden within approximately 8 - 10 hours at 77°F.

Precautions

(a) Fire Hazard: In connection with the original polyester or the sealing composition after combining polyester, plasticizer, and catalyst, the fire hazard involved is about the same as for an equal quantity of paint, and precautions should be taken as would be taken for paint in equal quantity and under similar conditions of application.

The catalyst is an organic peroxide which may detonate if it catches fire. Accordingly, the catalyst should be stored in small quantities and should be especially protected from open flames or other igniting agents such as red hot wires. After addition to the sealing composition, the catalyst is so diluted that it no longer constitutes a special hazard, and it does not increase the hazard of the mixture appreciably. During curing, the peroxide is entirely consumed and therefore disappears.

(b) Ventilation--Although polyesters have a moderately strong odor which is found by some persons to be seriously objectionable, the materials are not especially toxic. Ventilation equal to that when the same amount of paint is used should be adequate.

(c) Dermatitis-- Although polyesters have an odor, polyesters have not been found to cause dermatitis in general, even among operators who have their hands in the material much of the time. Accordingly, problems resulting from dermatitis should not be expected. However, it must be remembered that exposure to organic vapors or to volatile liquids constitutes abnormal exposure for the human skin, and, accordingly, it should be expected that an occasional person will be encountered who may have a more or less adverse reaction to these sealers.

Peroxides are strong oxidizing agents capable of causing severe burns to the unprotected skin. Accordingly, contact of skin with undiluted catalyst should be avoided.

Pot Life of Catalyzed Resin

The working life of Polyester A after mixing is approximately 2 - 3 hours at room temperature. For Polyester B under similar conditions, working life is approximately $2\frac{1}{2}$ to $3\frac{1}{2}$ hours. After this time, the sealer composition becomes too viscous for satisfactory spreading.

Cleaning of Utensils

Polyesters can be dissolved in most of the common hydrocarbon solvents such as toluene, benzene, acetone, etc, providing the resin has not gone beyond the gel stage. On the other hand, the cured, hard sealer is unaffected by any solvents. The hardened material is normally removed from its container by freezing. The resin contracts considerably during the freezing and can often be removed simply by inverting the container.

Storage Conditions

Even without the catalysts added, the polyesters will gradually polymerize at room temperatures and, after about 6-8 months at 75°F, will become too viscous for convenient use. Accordingly, it is preferable to store the polyesters at about 35°F or lower.

The catalyst itself is subject to slow decomposition at room temperature and, after about 3 months at 75°F, this decomposition will have seriously reduced the strength and activity of the catalyst. Accordingly, it is preferable to store the catalyst at 35°F or lower.

Appendix 2

Commercial Designations and Source of Recommended Polyesters and Catalyst Materials

Material	Characteristics	Source
Polyester A	Commercial Polyester, rigid type[1]	
Polyester E	Commercial Polyester, flexible type[1]	
Dimethyl phthalate	Plasticizer	Plastics Stock Rm.
Polybutadiene	Partially polymerized, liquid form	Philips Oil Company
Ethyl Lactate	Plasticizer	Chem Stock Rm.
Carbon Black	-	" " "
Bolts	Steel, 1/2" and 1" dia, N.C. - Class 2	Stores, Bldg 62
	Epoxy Resin	Houghton Labs
Catalyst	Methyl ethyl ketone peroxide[1]	

[1] See code sheet for trade name and commercial source.

TABLE 1

Effect of type of polyester on torque at room temperature

(a) Polyester A

1" dia bolts

Sample No.	Torque ft.-lbs.
1	460
2	470
3	450
4	460
5	420
6	470
7	450
Average	454

½" dia bolts

Sample No.	Torque ft.-lbs.
1	50
2	60
3	60
4	55
5	55
6	55
Average	56

(b) Polyester B

1" dia bolts

Sample No.	Torque ft.-lbs.
1	220
2	270
3	220
4	300
5	250
6	200
Average	243

½" dia bolts

Sample No.	Torque ft.-lbs.
1	31
2	31
3	32
4	32
5	
6	
Average	32

TABLE 2

Effect of Catalyst Concentration
Tested at Room Temperature on 1" Dia. Bolts

(a) Polyester A

Torque, ft-lbs

% by wt. of Lupersol DDM	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Average Torque Ft.-lbs
0.5	400	400				400
1.0	370	400				385
2.0	450	450				450
3.0	440	410				425

(b) Polyester B

% by wt of Lupersol DDM	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Average Torque Ft.-lbs
0.25	230	250	270			250
0.5	290	300	200	200	270	252
1.0	270	320				295
2.0	260	270	250	250	230	252
3.0	270	300				285
4.0	210	250	240			233

TABLE 3

Polyesters plus Polybutadiene (PBD)
Tested on 1" Dia Bolts

Torque, ft-lbs

(a) Polyester B

% by wt PBD	Sample 1	Sample 2	Sample 3	Sample 4	Average Torque Ft.-lbs
1	140	170	200		170
5	80	70	80		77
10	60	60	50		57
15	15	20	20		18
20	20	10	30		20

(b) Polyester A

% by wt PBD	Sample 1	Sample 2	Sample 3	Sample 4	Average Torque Ft.-lbs
1	250	300	270		273
5	160	180	170		170
10	140	100	100		113
15	50	80			65
20	50	40	60		50

TABLE 4

Polyesters Plus Polybutadiene (PBD)
Tested on $\frac{1}{2}$" Dia Bolts

(a) Polyester B

	Torque In.-lbs				Average Torque	
% by wt PBD	Sample 1	Sample 2	Sample 3	Sample 4	ft-lbs	in-lbs
1	215	100	65	125	11	126
5	80	60	55		5	65
10	35	35	35	40	3	36
15	30	20	45	15	2	28
20	10	10	15	35	1.5	18

(b) Polyester A

% by wt PBD	Sample 1	Sample 2	Sample 3	Sample 4	ft-lbs	in-lbs
1	315	325	375		28	338
5	275	225	230		20	243
10	150	150	75	200	12	144
15	75	50	70	85	6	70
20	25	60	25	30	3	35

TABLE 5

Polyesters Plus Dimethyl Phthalate (DMP)

A. Room Temperature

(1) Polyester B tested on 1" dia bolts.

% by wt. DMP	Torque Ft.-lbs 1	2	3	4	5	6	7	8	Average Torque Ft.-lbs
1	180	210	200	170	170	150			180
5	110	120	160	170	230	210			167
10	120	120	130	90	115	180	90		121
15	80	75	70	50	50	110	70		72
20	35	45	20	40	50	40	35		38
25	20	30	30	25					26

(2) Polyester A tested on 1" dia bolts.

% by wt. DMP	Torque Ft.-lbs 1	2	3	4	Average Torque Ft.-lbs
1	470	460	300	470	425
5	400	400	400	430	408
10	260	330	360	230	295
15	230		280		255
20	300	200	300	300	275
25	250	240	320	280	260

(3) Polyester B tested on ½" dia bolts.

% by wt. DMP	Torque Ft.-lbs 1	2	3	4	5	6	7	8	Average Torque Ft-lbs	Average Torque In-lbs
1	350	325	360	250	300	400	325		28	330
5	225	175	175	150	200	210	260	180	16	197
10	200	200	125	225	115	70	75	70	11	135
15	150	115	140	140	60	70	70	50	8	99
20	105	110	105	110	50	60	50	50	7	80
25	35	35	35	50					3	39

TABLE 5 (Contd)

(4) Polyester A tested on $\frac{1}{2}$" dia bolts.

% by wt. DMP	Torque Ft.-lbs 1	2	3	4	5	6	7	8	Average Torque Ft.-lbs	Average Torque In.-lbs
1	40	55	50						49	586
5	50	45	45	40					45	540
10	55	55	50	25					46	556
15	55	45	55	45					50	600
20	47	48	40	48					46	550
25	30	35	31	35					33	394

B. Tested at $\not<$ 160°F

(1) Polyester B tested on 1" dia bolts

% by wt. DMP	Torque Ft.-lbs						Average Torque Ft.-lbs
0	90	85	100	90	100	100	94
1	40	80	55	75			63
5	40	35	50	55			45
10	45	40	40	40			41
15	40	40	40	45			41
20	35	30	30	40			34
25	30	30	20	25			24

(2) Polyester A tested on 1" dia bolts.

% by wt. DMP	Torque Ft.-lbs						Average
0	440	430	420	475	390	380	423

TABLE 5 (Contd)

(3) Polyester B tested on ½" dia bolts

% by wt. DMP	Torque In.-lbs 1	2	3	4	5	6	7	8	Average Torque Ft-lbs	Average Torque In-lbs
0	80	75	80	100					7.0	84
1	80	80	75	75					6.5	78
5	50	30	75	40					4.1	49
10	25	25	25	25					2.0	25
15	20	20	20	20					1.8	21
20	20	10	20	20					1.5	18
25										

(4) Polyester A tested on ½" dia bolts

% by wt. DMP	Torque Ft.-lbs 1	2	3	4	Average Torque Ft-lbs	Average Torque In-lbs
0	50	35	35	40	40	480

C. Tested at $-65^{\circ}F$

(1) Polyester B tested on 1" dia bolts.

No reading could be obtained at $-65^{\circ}F$. This was due mainly to the difficulty of the operators in applying a constant rate of force on the torque wrench. At this temperature when torque is gradually applied the wrench would chatter and jerk to the extent that the indicator dial would vibrate vigorously.

(2) Polyester B tested on ½" dia bolts.

% by wt. DMP	Torque, Ft-lbs 1	2	3	4	Average Torque Ft-lbs	Average Torque In-lbs
1	60	50	40	30	45	540
5	30	60	70	40	50	600
10	40	50	55	30	44	526
15	55	55	35	20	41	496
20	30	45	20	25	30	360
25	35	35	50	50	43	510

TABLE 6

Vibration
Tested on 1" bolts

(a) $-65^{\circ}F$ Initial Temperature

	Torque Ft-lbs					Control (not vibrated)
Resin Composition	1	2	3	4	Av. Torque Ft-lbs.	Av. Torque Ft-lbs
Polyester A	310	500	525		445	454
Polyester B	250	300	210		253	243
Polyester B + 10% DMP	120	150	120		130	121
Polyester B + 10% PBD	30	40	20		30	57

(b) $+160^{\circ}F$ Initial Temperature

Polyester A	310	450	500		420	454
Polyester B	300	330	250		293	243
Polyester B + 10% DMP	100	110	130		113	121
Polyester B + 10% PBD	30	20	40		30	57

TABLE 7

Cycling
Tested on 1" dia bolts

	Torque Ft-lbs			
Resin Composition	1	2	3	Average Torque Ft-lbs
Polyester A	510	490	480	493
Polyester B	230	220	210	220
Polyester B + 10% DMP	150	150	100	133
Polyester B + 10% PBD	50	70	50	57

TABLE 8

Polyester Plus Carbon Black
Tested on 1" dia bolts

Torque Ft-lbs

(a) Polyester A

% C	1	2	3	Average Torque Ft-lbs
½	350	410	450	403
1	460	460	460	460

(b) Polyester B

½	260	250	300	270
1	250	280	270	267

TABLE 9

Polyester Plus Ethyl Lactate (E.L.)
Tested on 1" dia bolts

Torque Ft-lbs

(a) Polyester B

% by wt. E.L.	1	2	3	Average Torque Ft-lbs
1	260	330	290	293
5	200	190	140	176
10	100	100	110	103
20	40	20	20	27

(b) Polyester A

1	510	430	440	460
5	450	420	360	410
10	400	390	330	373

TABLE 10

Torque for 1" diameter, Class 2, National Coarse Bolts (3/4" Engagement)

Composition of Sealer	Torque (ft-lbs) at -65°F	+77°F	+160°F
Polyester A (rigid type)	> 550[1]	450 $\pm$ 40	425 $\pm$ 80
Polyester B (flexible type)	> 550[1]	240 $\pm$ 90	95 $\pm$ 15
Polyester B/dimethyl phthalate (99:1)	approx. 400[1]	180 $\pm$ 50	65 $\pm$ 40
" (95:5)	" 350[1]	170 $\pm$ 110	45 $\pm$ 20
" (90:10)	" 300[1]	120 $\pm$ 70	40 $\pm$ 5
" (85:15)	-[2]	70 $\pm$ 50	40 $\pm$ 5
" (75:25)	-[2]	25 $\pm$ 11	25 $\pm$ 12

[1] Available data not susceptible to statistical analysis.

[2] No data available.

TABLE 11

Torque for 1/2" diameter, Class 2, National Coarse Bolts (3/8" Engagement)

Composition of Sealer	Torque (ft-lbs) at -65°F	+77°F	+160°F
Polyester A (rigid type)	> 50[1]	55 $\pm$ 10	40 $\pm$ 1
Polyester B (flexible type)	> 50[1]	30 $\pm$ 2	7 $\pm$ 2.5
Polyester B/dimethyl phthalate (99:1)	45 $\pm$ 30	28 $\pm$ 20	6.5 $\pm$ 2.5
" (95:5)	50 $\pm$ 40	16[1]	4[1]
" (90:10)	45 $\pm$ 25	11[1]	2[1]
" (85:15)	40[1]	8[1]	2[1]
" (75:25)	40 $\pm$ 20	3 $\pm$ 1	-[2]

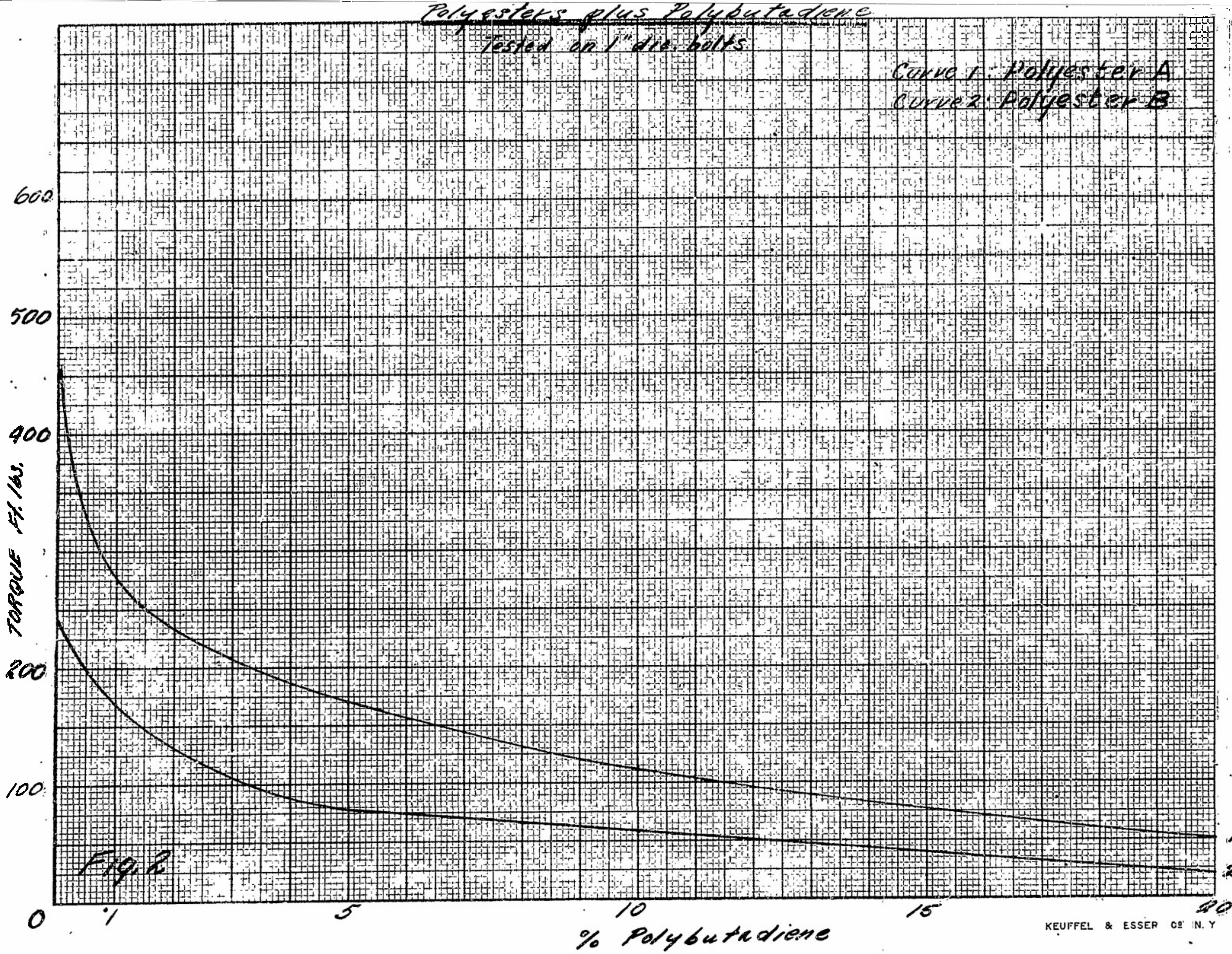
[1] Available data not susceptible to statistical analysis.

[2] No data available.

Effect of Catalyst Concentration

Tested on 1" dia. bolts

Fig. 1


Polyesters plus Polybutadiene
Tested on 1" dia. bolts
Curve 1: Polyester A
Curve 2: Polyester B
600
500
400
TORQUE Ft. lbs.
200
100
0
1
5
10
15
20
1
2
% Polybutadiene
KEUFFEL & ESSER CO. N.Y.

Polyester B + Polybutadiene

Tested on 1/2" dia. bolts

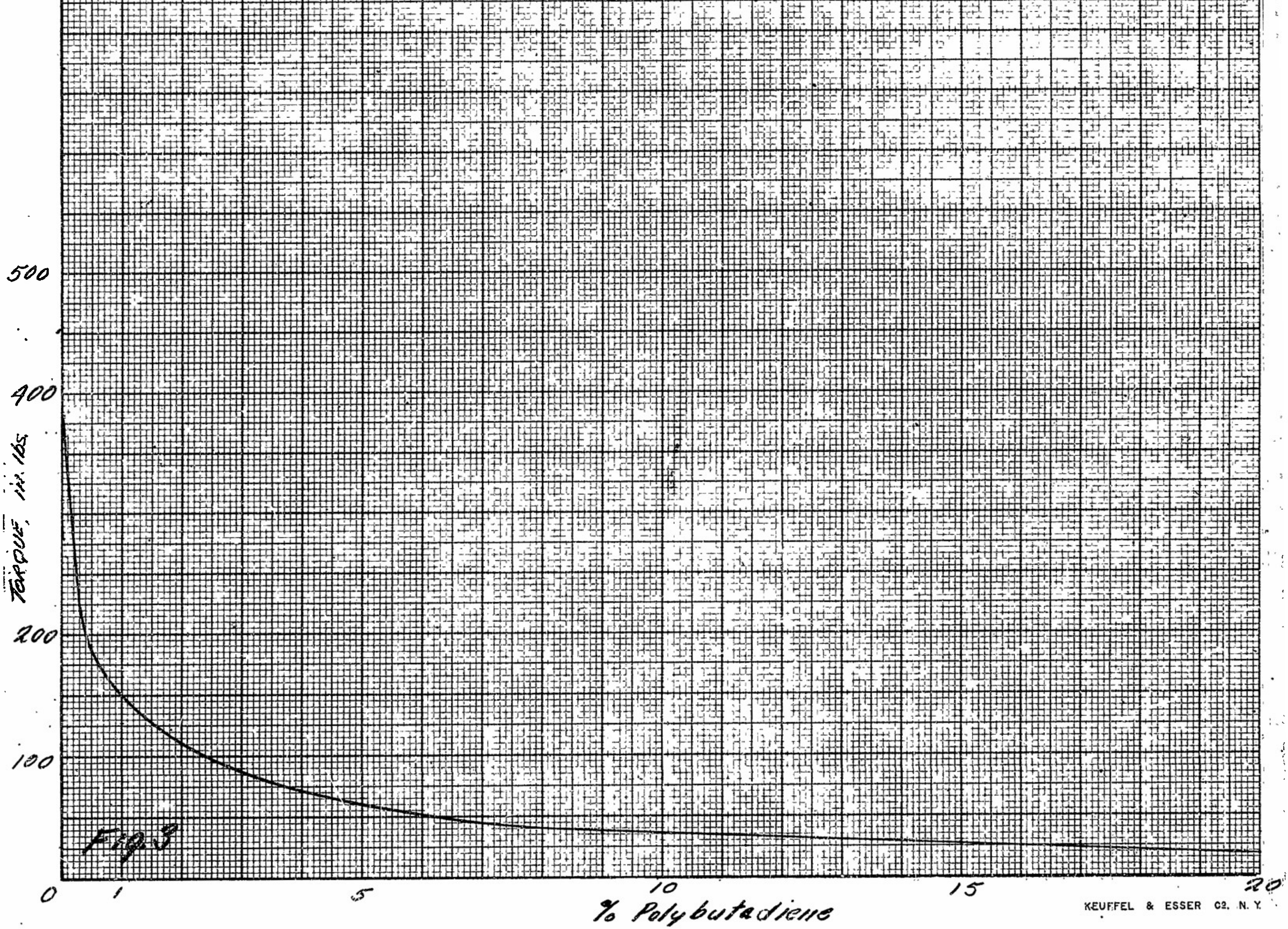


Fig. 3

Polyesters B + Dimethylphthalate
Tested on 1" dia. bolts

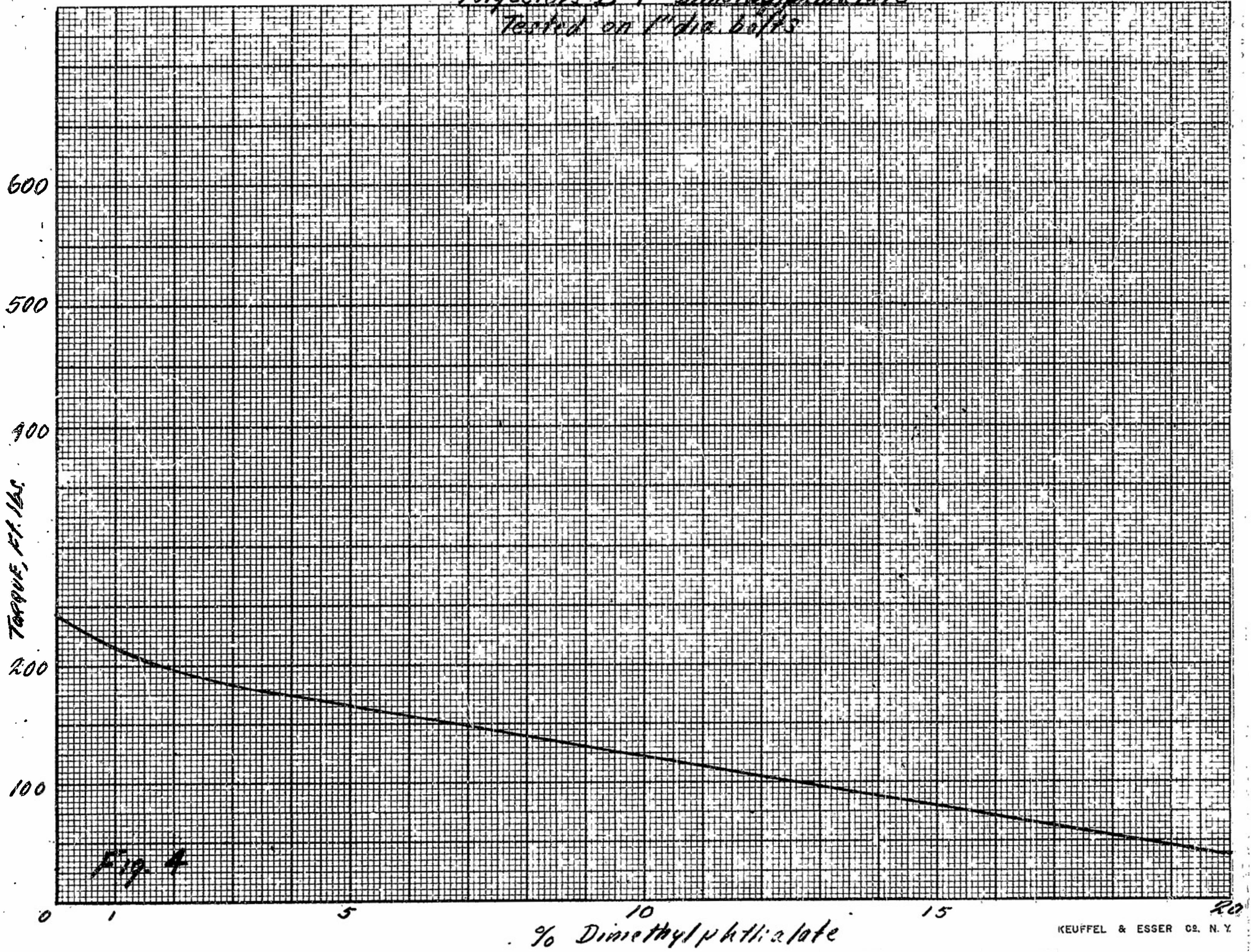
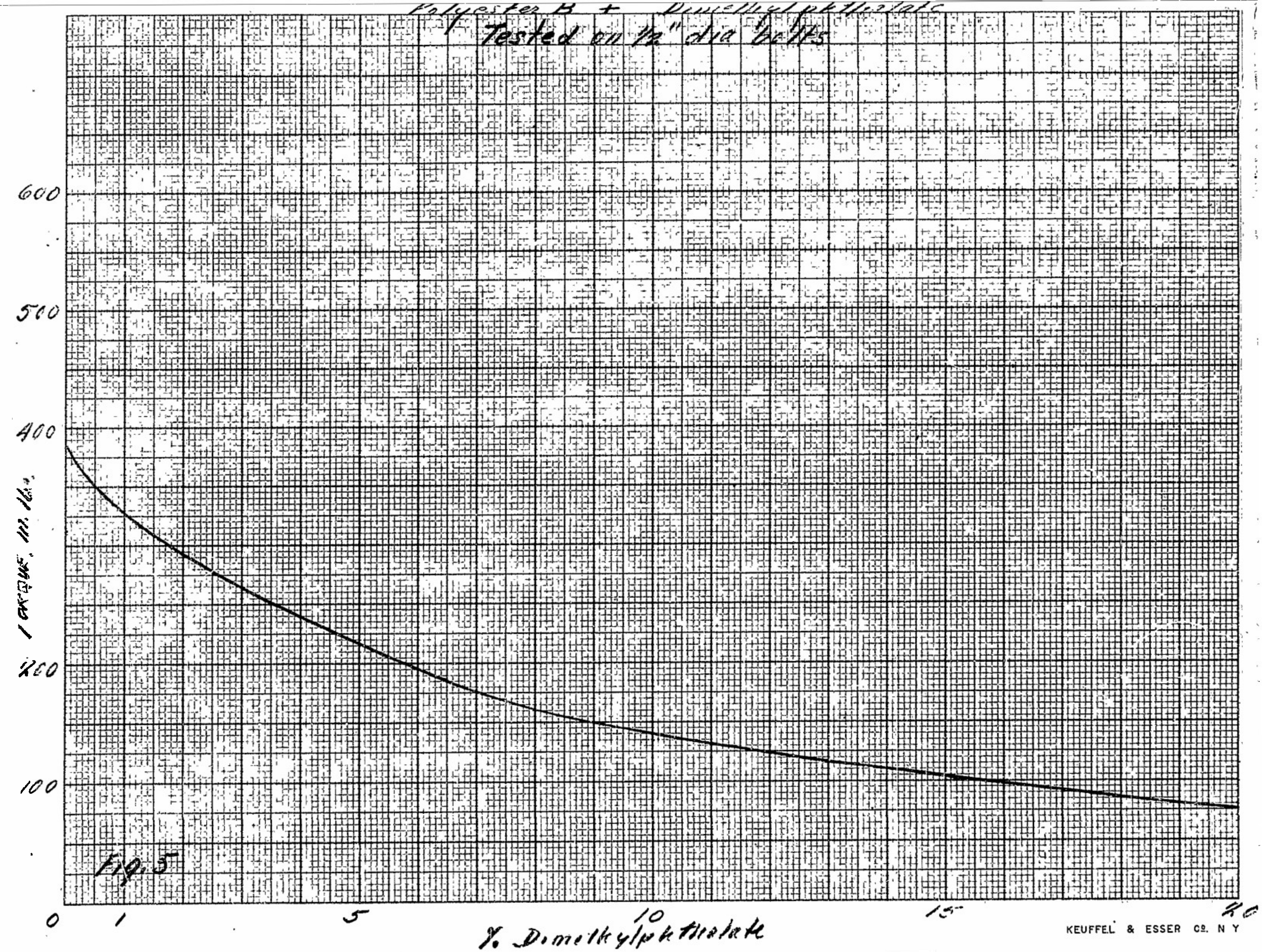
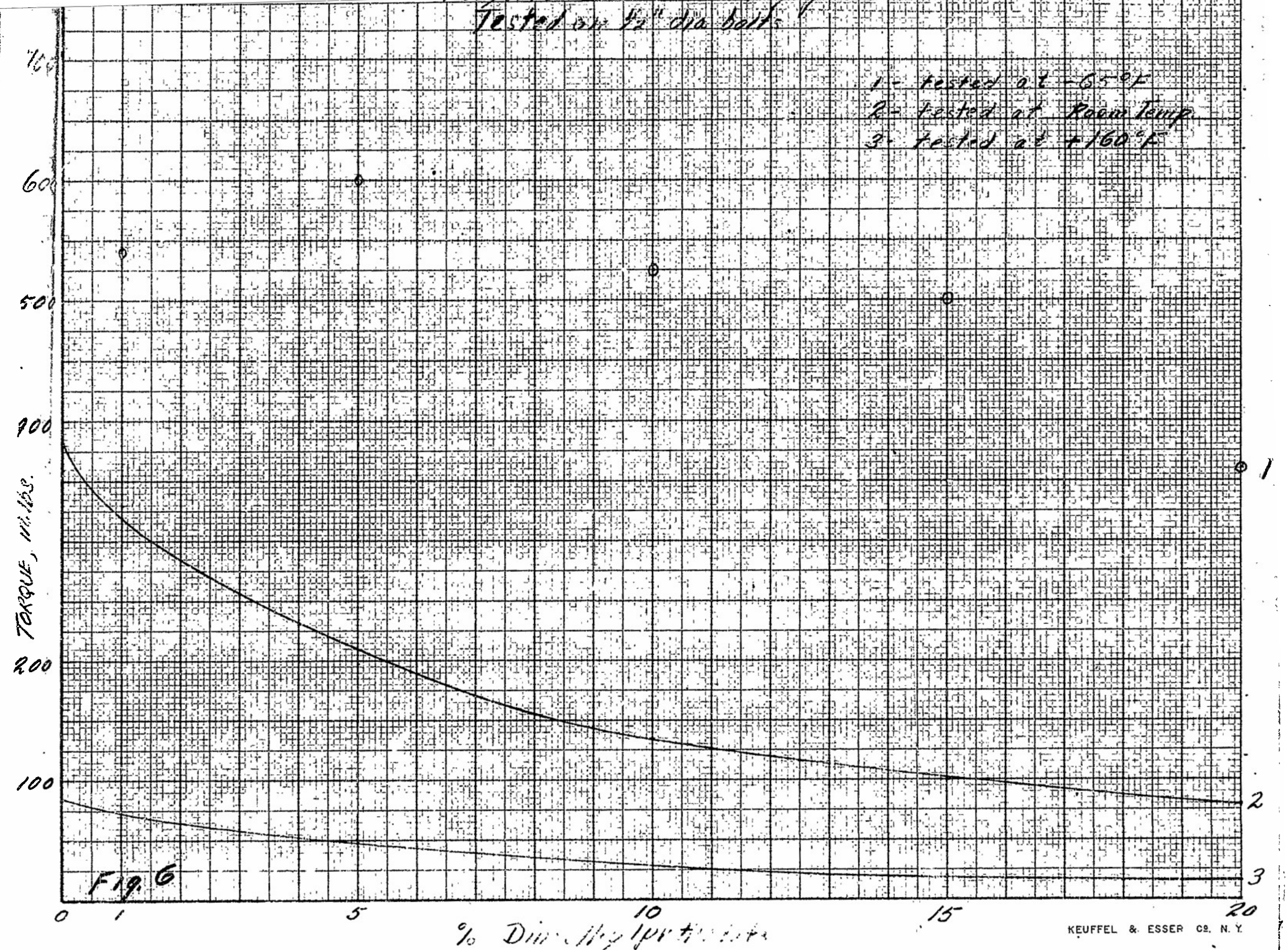


Fig. 4



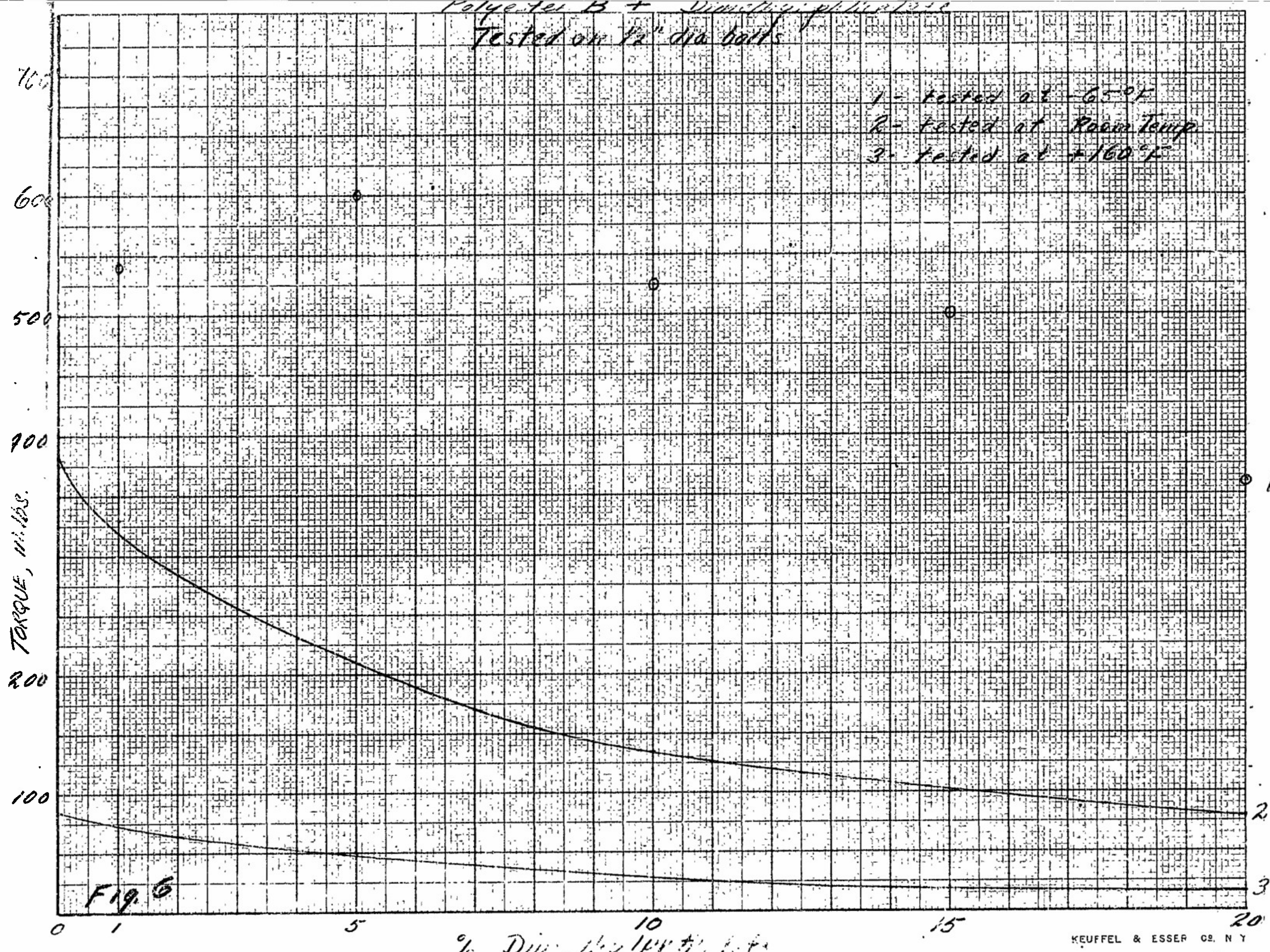
Polyester B + Dimethylphthalate
Tested on 1/2" dia bolts
600
500
400
200
100
0
1
5
10
15
20
Torque, in. lbs.
% Dimethylphthalate
KEUFFEL & ESSER CO. N Y

Fig. 5



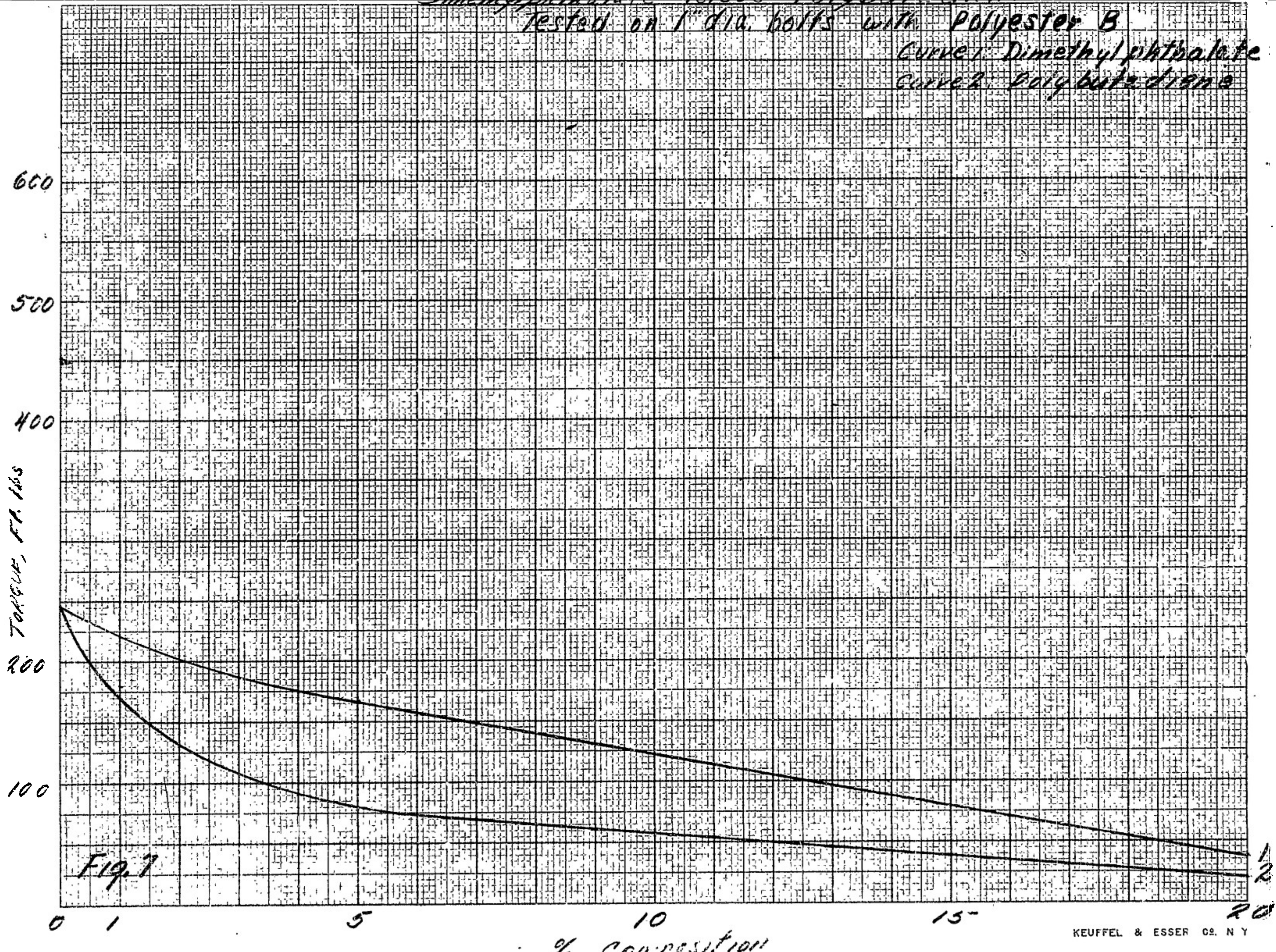
Tested on ½" dia bolts
1- tested at -65°F
2- tested at Room Temp
3- tested at +160°F
TORQUE, in.lbs.
100
200
400
500
600
0
1
5
10
15
20
1
2
3
KEUFFEL & ESSER CO. N.Y.

Fig. 6



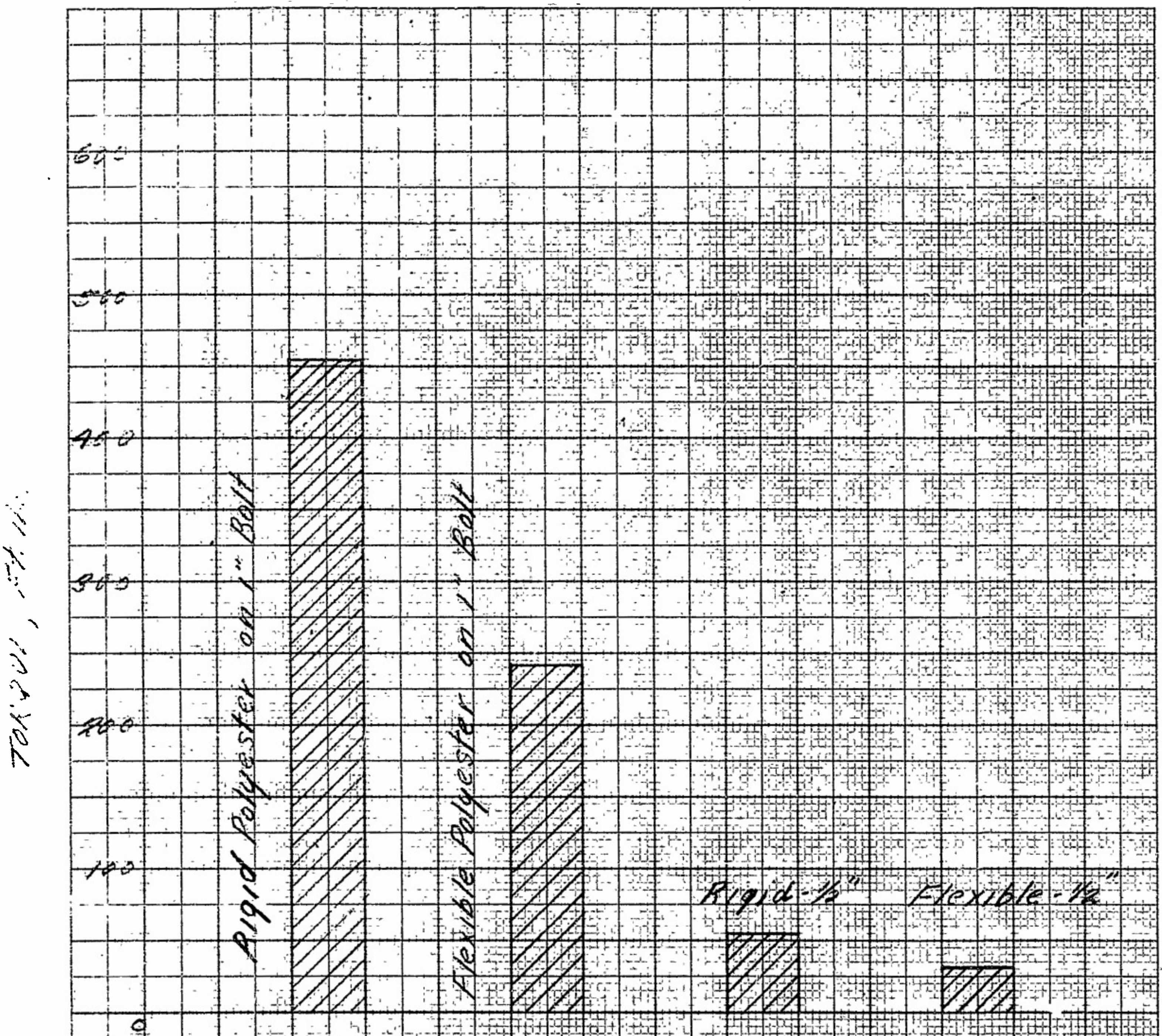
Polyester B +
Tested on 1/2" dia bolts
1- tested at -65°F
2- tested at Room Temp
3- tested at +160°F
TORQUE, in.lbs.
700
600
500
400
200
100
0
1
5
10
15
20
1
2
3
KEUFFEL & ESSER CO. N.Y.

Fig. 6



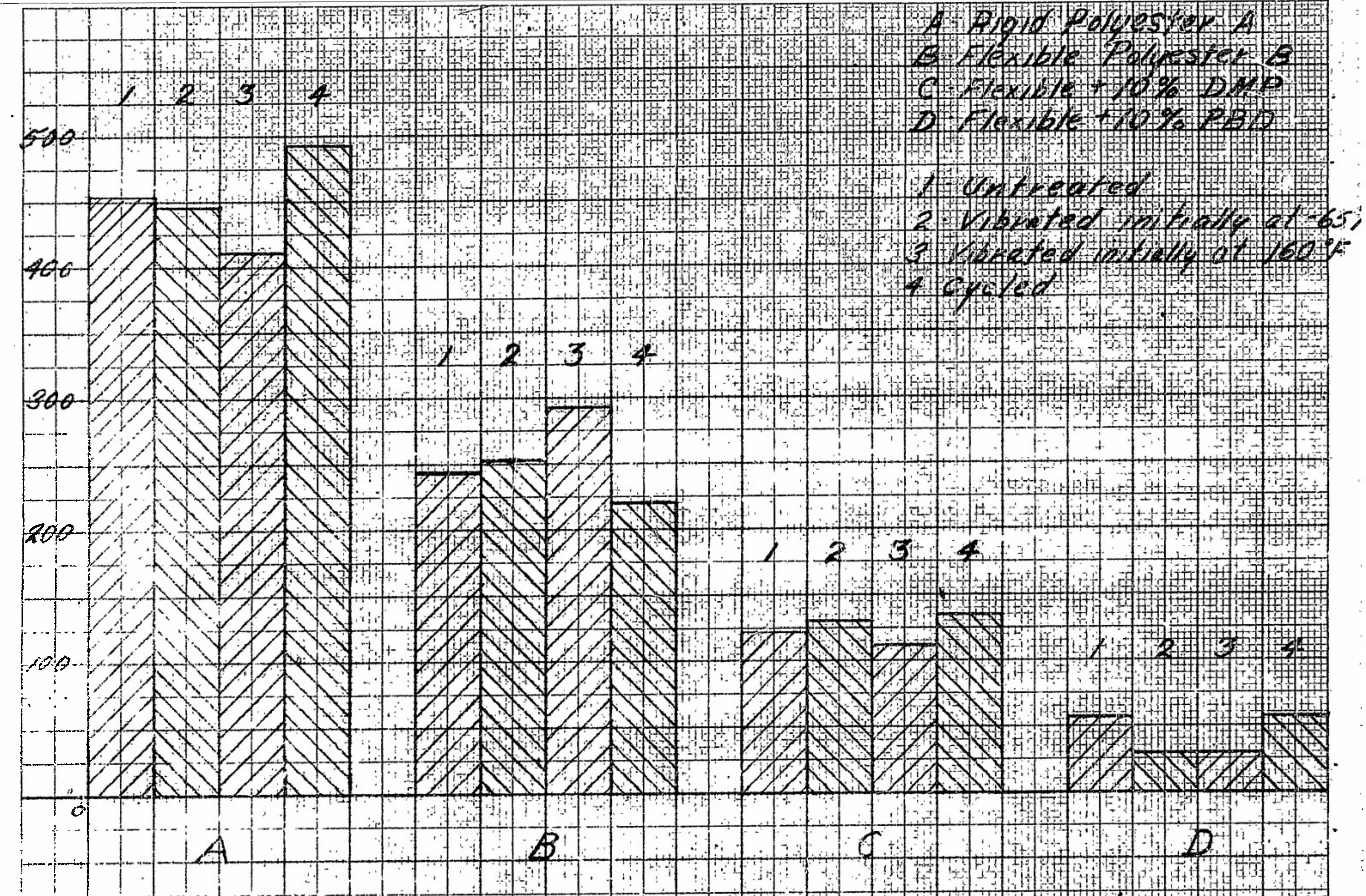
Tested on 1" dia. bolts with Polyester B
Curve 1: Dimethylphthalate
Curve 2: Polybutadiene
600
500
400
200
100
Torque, Ft. lbs
0
1
5
10
15
20
1
2
% Composition
KEUFFEL & ESSER CO. N Y

Fig. 1



The rigid type resin gives a higher torque than the flexible for any given bolt. Further, the one inch diameter bolt will have a higher torque than the one-half inch for any given resin.

Fig. 8



A Rigid Polyester A
B Flexible Polyester B
C Flexible + 10% DMP
D Flexible + 10% PBD
1 Untreated
2 Vibrated initially at -65°
3 Vibrated initially at 160°F
4 Cycled
500
400
300
200
100
0
1 2 3 4
A
B
C
D
Test performed on one inch diameter bolt

Fig 9